

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

United States Patent and Trademark  
Office  
(Box PCT)  
Crystal Plaza 2  
Washington, DC 20231  
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 24 March 1999 (24.03.99)	
International application No. PCT/SE98/01309	Applicant's or agent's file reference Case 581 PCT
International filing date (day/month/year) 03 July 1998 (03.07.98)	Priority date (day/month/year) 31 July 1997 (31.07.97)
Applicant LAMBERT, Nigel et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
16 February 1999 (16.02.99)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was  
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer A. Karkachi
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

# PATENT COOPERATION TREATY

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

## PCT

To:

Perstorp AB  
Patent Department  
284 80 PERSTORP

### NOTIFICATION OF RECEIPT OF DEMAND BY COMPETENT INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

(PCT Rules 59.3(e) and 61.1(b), first sentence  
and Administrative Instructions, Section 601(a))

Date of mailing  
(day/month/year)

17-02-1999

Applicant's or agent's file reference  
Case 581 PCT

#### IMPORTANT NOTIFICATION

International application No.

PCT/SE98/01309

International filing date (day/month/year)

03-07-1998

Priority date (day/month/year)

31-07-1997

Applicant  
Perstorp AB  
et al

1. The applicant is hereby notified that this International Preliminary Examining Authority considers the following date as the date of receipt of the demand for international preliminary examination of the international application:

16-02-1999

2. This date of receipt is:

- ☒ the actual date of receipt of the demand by this Authority (Rule 61.1(b)).
- ☐ the actual date of receipt of the demand on behalf of this Authority (Rule 59.3(e)).
- ☐ the date on which this Authority has, in response to the invitation to correct defects in the demand (Form PCT/IPEA/404), received the required corrections.

3. ☐ **ATTENTION:** That date of receipt is **AFTER** the expiration of 19 months from the priority date. Consequently, the election(s) made in the demand does (do) not have the effect of postponing the entry into the national phase until 30 months from the priority date (or later in some Offices) (Article 39(1)). Therefore, the acts for entry into the national phase must be performed within 20 months from the priority date (or later in some Offices) (Article 22). For details, see the *PCT Applicant's Guide*, Volume II.

- ☐ (If applicable) This notification confirms the information given by telephone, facsimile transmission or in person on:

4. Only where paragraph 3 applies, a copy of this notification has been sent to the International Bureau.

Name and mailing address of the IPEA/  
Patent- och registreringsverket  
Box 5055  
S-102 42 STOCKHOLM  
Facsimile No. 08-687 72 88

Telex  
17978  
PATOREG-S

Authorized officer

Telephone No. 08-782 25 00

**Inger Willén**

PCT

From the INTERNATIONAL BUREAU

NOTICE INFORMING THE APPLICANT OF THE  
COMMUNICATION OF THE INTERNATIONAL  
APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

To:

STENBERG, Yngve  
Perstorp AB  
S-284 80 Perstorp  
SUÈDE

Date of mailing (day/month/year) 11 February 1999 (11.02.99)		
Applicant's or agent's file reference Case 581 PCT		IMPORTANT NOTICE
International application No. PCT/SE98/01309	International filing date (day/month/year) 03 July 1998 (03.07.98)	
Applicant PERSTORP AB et al		

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:  
AU,BR,CN,EP,IL,JP,KP,KR,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AL,AM,AP,AT,AZ,BA,BB,BG,BY,CA,CH,CU,CZ,DE,DK,EA,EE,ES,FI,GB,GE,GH,GM,GW,HU,ID,IS,  
KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MD,MG,MK,MN,MW,MX,NO,NZ,OA,PL,PT,RO,RU,SD,SE,SG,SI,  
SK,SL,TJ,TM,TR,TT,UA,UG,UZ,VN,YU,ZW

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 11 February 1999 (11.02.99) under No. WO 99/06489

**REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)**

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

**REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))**

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer  J. Zahra
Facsimile No. (41-22) 740.14.35	Telephone No. (41-22) 338.83.38

## PATENT COOPERATION TREATY

PCT

From the INTERNATIONAL BUREAU

NOTIFICATION CONCERNING  
SUBMISSION OR TRANSMITTAL  
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

To:

STENBERG, Yngve  
Perstorp AB  
S-284 80 Perstorp  
SUEDE

Date of mailing (day/month/year) 19 August 1998 (19.08.98)	<b>IMPORTANT NOTIFICATION</b>
Applicant's or agent's file reference Case 581 PCT	
International application No. PCT/SE98/01309	
International publication date (day/month/year) Not yet published	
Applicant PERSTORP AB et al	

1. The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
2. This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
3. An asterisk(\*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
4. The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

<u>Priority date</u>	<u>Priority application No.</u>	<u>Country or regional Office or PCT receiving Office</u>	<u>Date of receipt of priority document</u>
31 July 1997 (31.07.97)	9716194.7	GB	06 Augu 1998 (06.08.98)

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Facsimile No. (41-22) 740.14.35

Authorized officer

Maria Victoria CORTIELLO

Telephone No. (41-22) 338.83.38

# PCT

## REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference  
(if desired) (12 characters maximum)

Case 581 PCT

### Box No. I TITLE OF INVENTION

A method of coating a substrate

### Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

Perstorp AB  
S-284 80 Perstorp  
Sweden

☐ This person is also inventor.

Telephone No.

+46 435 38000

Facsimile No.

+46 435 38100

Teleprinter No.

72000 perstp s

State (i.e. country) of nationality:  
SE

State (i.e. country) of residence:  
SE

This person is applicant  
for the purposes of:

☐ all designated  
States

☒ all designated States except  
the United States of America

☐ the United States  
of America only

☐ the States indicated in  
the Supplemental Box

### Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

Lambert, Nigel  
2 Collins Lane  
Ringwood  
Hants BH24 1LD  
England

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box  
is marked, do not fill in below.)

State (i.e. country) of nationality:  
GB

State (i.e. country) of residence:  
GB

This person is applicant  
for the purposes of:

☐ all designated  
States

☐ all designated States except  
the United States of America

☒ the United States  
of America only

☐ the States indicated in  
the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

### Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:

☒ agent

☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

Stenberg, Yngve  
c/o Perstorp AB  
S-284 80 Perstorp  
Sweden

Telephone No.

+46 435 38310

Facsimile No.

+46 435 38920

Teleprinter No.

72000 perstp s

☐ Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

## Continuation of Box No. III FURTHER APPLICANTS AND/OR (FURTHER) INVENTORS

If none of the following sub-boxes is used, this sheet is not to be included in the request.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

Strachan, Adrian  
9 Allumhurst Road  
Westbourne  
Bournemouth  
Dorset BH4 8EL  
England

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (i.e. country) of nationality:  
GB

State (i.e. country) of residence:  
GB

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☒ the United States of America only

☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

Wallis, Roger  
Willow Tree Cottage  
Waterditch  
Christchurch  
Dorset BH23 8JX  
England

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (i.e. country) of nationality:  
GB

State (i.e. country) of residence:  
GB

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☒ the United States of America only

☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

Wright, Vincent  
347A Lymington Road  
Highcliffe  
Hants BH23 5EK  
England

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (i.e. country) of nationality:  
GB

State (i.e. country) of residence:  
GB

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☒ the United States of America only

☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

This person is:

☐ applicant only

☐ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (i.e. country) of nationality:

State (i.e. country) of residence:

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☐ the United States of America only

☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

## Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

## Regional Patent

- ☒ AP ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ EA Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ EP European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ OA OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)

## National Patent (if other kind of protection or treatment desired, specify on dotted line):

- |  |  |
|--|--|
| <input checked="" type="checkbox"/> AL Albania                               | <input checked="" type="checkbox"/> LT Lithuania                                 |
| <input checked="" type="checkbox"/> AM Armenia                               | <input checked="" type="checkbox"/> LU Luxembourg                                |
| <input checked="" type="checkbox"/> AT Austria                               | <input checked="" type="checkbox"/> LV Latvia                                    |
| <input checked="" type="checkbox"/> AU Australia                             | <input checked="" type="checkbox"/> MD Republic of Moldova                       |
| <input checked="" type="checkbox"/> AZ Azerbaijan                            | <input checked="" type="checkbox"/> MG Madagascar                                |
| <input checked="" type="checkbox"/> BA Bosnia and Herzegovina                | <input checked="" type="checkbox"/> MK The former Yugoslav Republic of Macedonia |
| <input checked="" type="checkbox"/> BB Barbados                              | <input checked="" type="checkbox"/> MN Mongolia                                  |
| <input checked="" type="checkbox"/> BG Bulgaria                              | <input checked="" type="checkbox"/> MW Malawi                                    |
| <input checked="" type="checkbox"/> BR Brazil                                | <input checked="" type="checkbox"/> MX Mexico                                    |
| <input checked="" type="checkbox"/> BY Belarus                               | <input checked="" type="checkbox"/> NO Norway                                    |
| <input checked="" type="checkbox"/> CA Canada                                | <input checked="" type="checkbox"/> NZ New Zealand                               |
| <input checked="" type="checkbox"/> CH and LI Switzerland and Liechtenstein  | <input checked="" type="checkbox"/> PL Poland                                    |
| <input checked="" type="checkbox"/> CN China                                 | <input checked="" type="checkbox"/> PT Portugal                                  |
| <input checked="" type="checkbox"/> CU Cuba                                  | <input checked="" type="checkbox"/> RO Romania                                   |
| <input checked="" type="checkbox"/> CZ Czech Republic                        | <input checked="" type="checkbox"/> RU Russian Federation                        |
| <input checked="" type="checkbox"/> DE Germany                               | <input checked="" type="checkbox"/> SD Sudan                                     |
| <input checked="" type="checkbox"/> DK Denmark                               | <input checked="" type="checkbox"/> SE Sweden                                    |
| <input checked="" type="checkbox"/> EE Estonia                               | <input checked="" type="checkbox"/> SG Singapore                                 |
| <input checked="" type="checkbox"/> ES Spain                                 | <input checked="" type="checkbox"/> SI Slovenia                                  |
| <input checked="" type="checkbox"/> FI Finland                               | <input checked="" type="checkbox"/> SK Slovakia                                  |
| <input checked="" type="checkbox"/> GB United Kingdom                        | <input checked="" type="checkbox"/> SL Sierra Leone                              |
| <input checked="" type="checkbox"/> GE Georgia                               | <input checked="" type="checkbox"/> TJ Tajikistan                                |
| <input checked="" type="checkbox"/> GH Ghana                                 | <input checked="" type="checkbox"/> TM Turkmenistan                              |
| <input checked="" type="checkbox"/> GM Gambia                                | <input checked="" type="checkbox"/> TR Turkey                                    |
| <input checked="" type="checkbox"/> GW Guinea-Bissau                         | <input checked="" type="checkbox"/> TT Trinidad and Tobago                       |
| <input checked="" type="checkbox"/> HU Hungary                               | <input checked="" type="checkbox"/> UA Ukraine                                   |
| <input checked="" type="checkbox"/> ID Indonesia                             | <input checked="" type="checkbox"/> UG Uganda                                    |
| <input checked="" type="checkbox"/> IL Israel                                | <input checked="" type="checkbox"/> US United States of America                  |
| <input checked="" type="checkbox"/> IS Iceland                               | <input checked="" type="checkbox"/> UZ Uzbekistan                                |
| <input checked="" type="checkbox"/> JP Japan                                 | <input checked="" type="checkbox"/> VN Viet Nam                                  |
| <input checked="" type="checkbox"/> KE Kenya                                 | <input checked="" type="checkbox"/> YU Yugoslavia                                |
| <input checked="" type="checkbox"/> KG Kyrgyzstan                            | <input checked="" type="checkbox"/> ZW Zimbabwe                                  |
| <input checked="" type="checkbox"/> KP Democratic People's Republic of Korea |  |
| <input checked="" type="checkbox"/> KR Republic of Korea                     |  |
| <input checked="" type="checkbox"/> KZ Kazakhstan                            |  |
| <input checked="" type="checkbox"/> LC Saint Lucia                           |  |
| <input checked="" type="checkbox"/> LK Sri Lanka                             |  |
| <input checked="" type="checkbox"/> LR Liberia                               |  |
| <input checked="" type="checkbox"/> LS Lesotho                               |  |

Check-boxes reserved for designating States (for the purposes of a national patent) which have become party to the PCT after issuance of this sheet:

☐

☐

☐

In addition to the designations made above, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except the designation(s) of \_\_\_\_\_

The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)

## Box No. VI PRIORITY CLAIM

Further priority claims are indicated in the Supplemental Box ☐

The priority of the following earlier application(s) is hereby claimed:

Country (in which, or for which, the application was filed)	Filing Date (day/month/year)	Application No.	Office of filing (only for regional or international application)
item (1) England	31 July 1997 31 - 07 - 1997	9716194.7	
item (2)			
item (3)			

Mark the following check-box if the certified copy of the earlier application is to be issued by the Office which for the purposes of the present international application is the receiving Office (a fee may be required):

☐ The receiving Office is hereby requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s): \_\_\_\_\_

## Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) (If two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used): ISA / \_\_\_\_\_

Earlier search Fill in where a search (international, international-type or other) by the International Searching Authority has already been carried out or requested and the Authority is now requested to base the international search, to the extent possible, on the results of that earlier search. Identify such search or request either by reference to the relevant application (or the translation thereof) or by reference to the search request:

Country (or regional Office): \_\_\_\_\_

Date (day/month/year): \_\_\_\_\_

Number: \_\_\_\_\_

## Box No. VIII CHECK LIST

This international application contains the following number of sheets:

1. request : 4 sheets  
2. description : 17 sheets  
3. claims : 3 sheets  
4. abstract : 1 sheets  
5. drawings : 3 sheets

Total : 28 sheets

This international application is accompanied by the item(s) marked below:


1. ☐ separate signed power of attorney  
2. ☒ copy of general power of attorney  
3. ☐ statement explaining lack of signature  
4. ☐ priority document(s) identified in Box No. VI as item(s): \_\_\_\_\_  
5. ☒ fee calculation sheet  
6. ☐ separate indications concerning deposited microorganisms  
7. ☐ nucleotide and/or amino acid sequence listing (diskette)  
8. ☐ other (specify): \_\_\_\_\_

Figure No. \_\_\_\_\_ of the drawings (if any) should accompany the abstract when it is published.

## Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request):

Perstorp July 1, 1998  
PERSTORP AB

  
Yngve Stenberg

For receiving Office use only

1. Date of actual receipt of the purported international application: \_\_\_\_\_

3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application: \_\_\_\_\_

4. Date of timely receipt of the required corrections under PCT Article 11(2): \_\_\_\_\_

5. International Searching Authority specified by the applicant: ISA / \_\_\_\_\_

6. ☐ Transmittal of search copy delayed until search fee is paid

2. Drawings:

☐ received:☐ not received:

For International Bureau use only

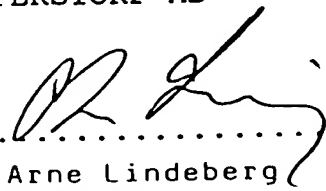
Date of receipt of the record copy  
by the International Bureau: \_\_\_\_\_

GENERAL AUTHORIZATION

A worldwide general authorization for the Patent Council and Officer of our patent department Yngve Gunnar William Stenberg or anyone that he may appoint in his stead, to file on our behalf applications on patents, trade marks and designs, to lodge oppositions against such applications belonging to others and to sign on our behalf letters of consent (Zustimmungserklärungen) relating to inquiries from others about consent to register their trade marks, that in everything concerning our applications as well as patents, trade mark registrations and design registrations granted on said applications and our oppositions, act on our behalf by signing Declarations of all kinds comprising novelty declarations relating to our design applications and counter declarations (Zustimmungserklärungen) relating to our trade mark applications, and to withdraw such applications at any time after filing, and at any time to inspect and obtain copies of all documents concerning such applications; declaring ourselves satisfied with such legal actions as our Patent Council and Officer may take to this effect.

Perstorp, October 18th 1991

PERSTORP AB

  
.....  
Arne Lindeberg  
Direktör

  
.....  
Mats Tunér  
Direktör

# PCT

## FEE CALCULATION SHEET

Annex to the Request

For receiving Office use only

International application No.

Date stamp of the receiving Office

Applicant's or agent's  
file reference

Case 581 PCT

Applicant

Perstorp AB  
S-284 80 Perstorp  
Sweden

### CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE

1000

T

2. SEARCH FEE

6800

S

International search to be carried out by

(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FEE

Basic Fee

The international application contains 28 sheets.

first 30 sheets

3500

b<sub>1</sub>

remaining sheets

additional amount

3500

b<sub>2</sub>

Add amounts entered at b<sub>1</sub> and b<sub>2</sub> and enter total at B

3500

B

Designation Fees

The international application contains designations.

11

800

8800

D

number of designation fees payable (maximum 11) amount of designation fee

Add amounts entered at B and D and enter total at I

12300

I

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicant is (or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT

P

5. TOTAL FEES PAYABLE

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

20100

TOTAL

☐ The designation fees are not paid at this time.

### MODE OF PAYMENT

☐ authorization to charge  
deposit account (see below)

☐ cheque

☐ postal money order

☐ bank draft

☐ cash

☐ revenue stamps

☐ coupons

☒ other (specify): Bank giro

### DEPOSIT ACCOUNT AUTHORIZATION (this mode of payment may not be available at all receiving Offices)

The RO/

☐

is hereby authorized to charge the total fees indicated above to my deposit account.

☐

is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

☐

is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

Deposit Account Number

Date (day/month/year)

Signature

## PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

REC'D 14 DEC 1999

WIPO

PCT

Applicant's or agent's file reference Case 581 PCT	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/SE98/01309	International filing date (day/month/year) 03.07.1998	Priority date (day/month/year) 31.07.1997
International Patent Classification (IPC) or national classification and IPC <sub>7</sub> C 09 D 4/00 // C 08 F 2/48		
Applicant Perstorp AB et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand  16.02.1999	Date of completion of this report  22.11.1999
Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. 08-667 72 88	Authorized officer  Barbro Nilsson/EÖ Telephone No. 08-782 25 00

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/SE98/01309

## I. Basis of the report

1. This report has been drawn on the basis of *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.)*:

- ☐ the international application as originally filed.
- ☒ the description, pages 1-17, as originally filed,  
 pages \_\_\_\_\_, filed with the demand,  
 pages \_\_\_\_\_, filed with the letter of \_\_\_\_\_,  
 pages \_\_\_\_\_, filed with the letter of \_\_\_\_\_.
- ☒ the claims, Nos. \_\_\_\_\_, as originally filed,  
 Nos. \_\_\_\_\_, as amended under Article 19,  
 Nos. \_\_\_\_\_, filed with the demand,  
 Nos. 1-22, filed with the letter of 16.09.1999,  
 Nos. \_\_\_\_\_, filed with the letter of \_\_\_\_\_.
- ☒ the drawings, sheets/fig \_\_\_\_\_, as originally filed,  
 sheets/fig \_\_\_\_\_, filed with the demand  
 sheets/fig 4, filed with the letter of 16.09.1999,  
 sheets/fig \_\_\_\_\_, filed with the letter of \_\_\_\_\_.

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages \_\_\_\_\_
- ☒ the claims, Nos. 23
- ☐ the drawings, sheets/fig \_\_\_\_\_

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the supplemental Box (Rule 70.2(c)).

4. Additional observations, if necessary:

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/SE98/01309

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement****1. Statement**

Novelty (N)	Claims	<u>1-22</u>	YES
	Claims		NO
Inventive step (IS)	Claims	<u>1-22</u>	YES
	Claims		NO
Industrial applicability (IA)	Claims	<u>1-22</u>	YES
	Claims		NO

**2. Citations and explanations**

The present invention relates to a method of coating a substrate and to the said substrate.

The invention deals with the problem of curing a composition comprising multi-functional material with ultra-violet light. Normally curing is performed in the presence of a photo-initiator. However, according to this invention a composition comprising a mixture including at least a reactive part comprising between 30% and 100% multi-functional material can be cured when exposed to UV-light without the need of a photo-initiator. A substantially inert atmosphere is maintained in the curing zone when the substrate is exposed to UV-light.

The most relevant document cited in the International Search Report is US 5047261. This document discloses photo-crosslinking in UV-light without the need of a photo-initiator, even though employing a monoacrylic reactive diluent. It is further disclosed that polyfunctional acrylates enable the reactivity to be increased in comparison with the use of monofunctional acrylates, see column 2, lines 3-8. However, it is also disclosed that the use of polyfunctional acrylates results in a residual unsaturated content, which is markedly higher after radio cross-linking. This results in a less satisfactory behaviour of the coating towards light during ageing, in that it yellows rapidly with possible losses of mechanical properties, column 2, lines 10-17. Thus, the cited US patent teaches that such polyfunctional acrylates should be avoided. Moreover, the document does not mention anything about an inert atmosphere, which is necessary according to the present invention. In the light of these facts the claimed invention can not be considered obvious to a person skilled in the art.

.../...

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/SE98/01309

**Supplemental Box**

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: V

Therefore, the invention claimed is novel, is considered to involve an inventive step and to have industrial applicability.

16-09-1999

-18-

CLAIMS:

1. A method of coating a substrate, the method comprising the steps of applying a coating composition to at least selected areas of the substrate, exposing the coated substrate to ultra-violet light from at least one lamp having a power, output of at least 140 watts per linear centimetre in a curing zone, to initiate curing of the coating, the coating composition comprising a mixture including at least a reactive part comprising between 30% and 100% multi-functional material and being photo-initiator-free, including the step of maintaining a substantially inert atmosphere in the curing zone where the substrate is exposed to said ultra-violet light.
2. A method according to Claim 1 wherein the inert atmosphere is obtained by purging the said curing zone with inert gas.
3. A method according to Claim 2 wherein the inert gas comprises nitrogen.
4. A method according to any one of the preceding Claims wherein the oxygen concentration within the said curing zone is less than 1,000 parts per million.
5. A method according to Claim 4 wherein the oxygen concentration is less than 100 parts per million.
6. A method according to any one of the preceding Claims wherein the multi-functional material comprises one or more reactive diluents.
7. A method according to any one of the preceding Claims wherein the multi-functional material comprises one or more

AMENDED SHEET

16-09-1999

-19-

materials, the or each material having a molecular weight in excess of 480.

8. A method according to any one of the preceding Claims wherein the multi-functional material comprises one or more materials which have three or more functional acrylate groups.

9. A method according to Claim 6, 7 or 8 wherein the coating material additionally contains a pre-polymer.

10. A method according to Claim 9 wherein the pre-polymer comprises polyester acrylate, polyurethane acrylate, epoxyacrylate, or a full acrylate material.

11. A method according to Claim 9 or 10 wherein the pre-polymer is multi-functional.

12. A method according to any one of the preceding Claims wherein the coating composition comprises, in addition to the reactive part, a filler.

13. A method according to Claim 12 wherein the filler is clay.

14. A method according to Claim 12 wherein the filler is silica.

15. A method according to Claim 12 wherein the filler is magnetisable particles.

16. A method according to any one of the preceding Claims wherein the power output of the lamp is at least 180 watts/cm.

AMENDED SHEET

16 -09- 1999

-20-

17. A method according to Claim 16 wherein the power output of the lamp is substantially 240 watts/cm.

18. A method according to any one of the preceding Claims wherein UV light from the lamp has a substantial spectral content in the range of 200-300 nm.

19. A method according to Claim 18 wherein UV light from the lamp has a spectral content at peaks of approximately 370 nm, 408 nm and 438 nm.

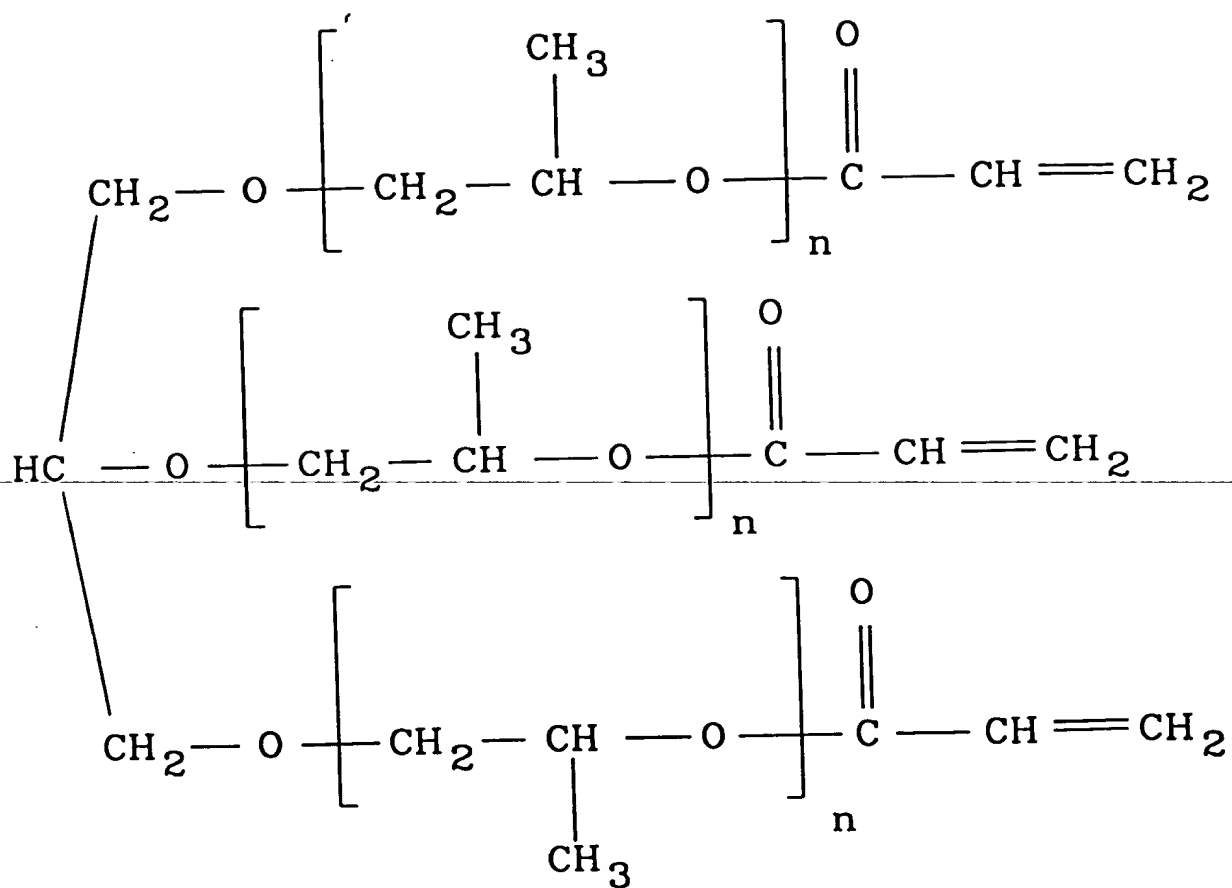
20. A method according to any one of the preceding Claims wherein two lamps are provided in the curing zone, the lamps having different spectral properties.

21. A method according to any one of Claims 1 to 19 wherein two lamps are provided in the curing zone, the lamps having substantially identical spectral properties.

22. A substrate when coated by a method according to any one of the preceding Claims.

AMENDED SHEET

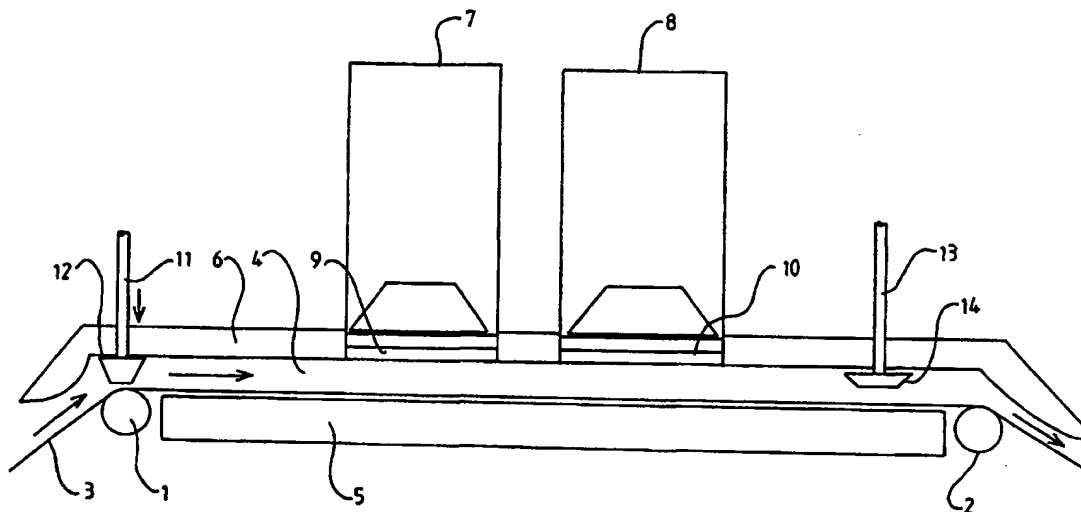
3 / 3

FIG 4

## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : C09D 4/00 // C08F 2/48		A1	(11) International Publication Number: WO 99/06489
		(43) International Publication Date: 11 February 1999 (11.02.99)	
(21) International Application Number: PCT/SE98/01309 (22) International Filing Date: 3 July 1998 (03.07.98) (30) Priority Data: 9716194.7 31 July 1997 (31.07.97) GB (71) Applicant (for all designated States except US): PERSTORP AB [SE/SE]; S-284 80 Perstorp (SE). (72) Inventors; and (75) Inventors/Applicants (for US only): LAMBERT, Nigel [GB/GB]; 2 Collins Lane, Ringwood, Hants BH24 1LD (GB). STRACHAN, Adrian [GB/GB]; 9 Allumhurst Road, Westbourne, Bournemouth, Dorset BH4 8EL (GB). WAL-LIS, Roger [GB/GB]; Willow Tree Cottage, Waterditch, Christchurch, Dorset BH23 8JX (GB). WRIGHT, Vincent [GB/GB]; 347A Lymington Road, Highcliffe, Hants BH23 5EK (GB). (74) Agent: STENBERG, Yngve; Perstorp AB, S-284 80 Perstorp (SE).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  Published With international search report.	

(54) Title: A METHOD OF COATING A SUBSTRATE



## (57) Abstract

A method of coating a substrate comprises the steps of applying a coating composition to selected areas of the substrate. The coating composition comprises a mixture including at least a reactive part. The reactive part comprises between 30 % and 100 % multi-functional material, and is photoinitiator free. The coated substrate is exposed, in a curing zone, to ultra-violet light from at least one lamp which has a power output of at least 140 wats per linear centimetre. The ultra-violet light initiates curing of the coating. A substantially inert atmosphere is maintained in the curing zone where the substrate is exposed to the ultra-violet light.

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Larvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

## A METHOD OF COATING A SUBSTRATE

The present invention relates to a method of coating a substrate.

It has been proposed previously to provide various coatings which can be applied to a substrate in a liquid form and which can then be cured to form a solid coating. Typically, the liquid coating incorporates unsaturated organic compounds which include C=C double bonds. These compounds present within the liquid coating are effectively polymerised during the curing process.

It has been proposed to effect the curing utilising high energy electron radiation. Typically electrons are delivered by an electron beam accelerator which normally operates at a voltage in excess of 150kVe, although alternatively a nuclear source may be utilised. The radiation breaks some of the C=C double bonds present in the unsaturated organic material, generating free radicals which initiate free radical polymerisation of the remaining material. The equipment necessary to carry out this process is costly to purchase and has to be specially shielded to avoid any leakage of gamma radiation.

There have been many proposals concerning coatings which can be cured, in response to ultra-violet light, involving free radical initiated polymerisation. Typically these coatings utilise a photo-initiator. A photo-initiator in this process is a material that absorbs light,

-2-

and generates free radicals. The free radicals initiate the polymerisation of the coating. Photo-initiators are generally expensive, and can give rise to problems. For example, they can create an undesirable odour or taste (which may be relevant when the coating is going to be in contact with a food product) and can also cause "yellowing", which is a tendency for the cured coating to adopt a yellow colour over the course of time.

It has been proposed to provide a coating which can be cured on exposure to ultra-violet light which does not incorporate a photo-initiator. A coating of this type is disclosed in US-A-5,446,073. This Specification teaches a formulation which has a balance of "acceptor" and "donor" species. The process described in US-A-5,446,073 has not yet been adopted by industry, primarily because the reactive materials required are not readily commercially available. ~~Also, the curing process is relatively slow~~ with cure times typically being measured in terms of minutes. A further disadvantage of this technique is that it requires a combination of electron donating monomers and electron accepting monomers of relatively low molecular weight, and monomers in general are regarded as being prone to shrinkage during cure and are also regarded as being toxic since they may relatively easily penetrate the skin.

It has been discovered that short wavelength light may be used to effect a cure by direct fragmentation, in a similar way to the electron beam accelerator. Thus, it has been proposed to use light from excimer lamps, which have a wavelength of 172 nm, to cure radiation curable coatings without the use of a photo-initiator. However, this technique has only been used successfully with very thin coatings, typically coatings less than 1  $\mu\text{m}$  thick. The excimer energy is not able to penetrate readily into a

-3-

coating which is of a greater thickness without excessive heat being generated. If an excimer lamp were used to irradiate a 10  $\mu$ m thick coating, it would produce a "cured skin" on the surface, but not a complete cure.

The present invention seeks to provide a UV cured coating in which the disadvantages of prior proposals are obviated or reduced.

According to one aspect of the present invention, there is provided a method of coating a substrate, the method comprising the steps of applying a coating composition to at least selected areas of the substrate, exposing the coated substrate to ultra-violet light from at least one lamp having a power output of at least 140 watts per linear centimetre in a curing zone, to initiate curing of the coating, the coating composition comprising a mixture including at least a reactive part comprising between 30% and 100% multi-functional material and being photo-initiator-free, including the step of maintaining a substantially inert atmosphere in the curing zone where the substrate is exposed to said ultra-violet light.

The preferred multi-functional materials have a functionality of at least three.

Preferably, the inert atmosphere is obtained by purging the curing zone with inert gas such as nitrogen.

Advantageously, the oxygen concentration in the curing zone is less than 1,000 ppm and preferably less than 100 ppm.

Preferably, the multi-functional material comprises one or more reactive diluents.

-4-

Conveniently, the multi-functional material comprises one or more materials, each material having a molecular weight in excess of 480.

Advantageously, the multi-functional material comprises one or more materials which have three or more functional acrylate groups.

Conveniently, the coating material contains a pre-polymer, and may comprise polyester acrylate, polyurethane acrylate, epoxyacrylate or a full acrylic material.

Conveniently, the pre-polymer is multi-functional.

Advantageously, the coating composition comprises, in addition to the reactive part, a filler, and the filler may comprises ~~clay, silica or magnetisable particles.~~

Preferably, the power output of the lamp is at least 180 watts/cm and may be substantially 240 watts/cm.

Conveniently, UV light from the lamp has a substantial spectral content in the range 200-300 nm.

Preferably, UV light from the lamp has additional spectral content with peaks of approximately 370 nm, 408 nm and 438 nm.

Two or more lamps may be provided in the curing zone. The lamps may have different spectral properties or may have substantially identical spectral properties.

The invention relates to a substrate when coated by a

-5-

method as described above.

In order that the invention may be more readily understood, and so that further features thereof may be appreciated, the invention will now be described, by way of example, with reference to the accompanying drawings in which:

FIGURE 1 is a diagrammatic view of an apparatus for use in curing a coating on a substrate;

FIGURE 2 is a graphical indication of the spectral output of a preferred UV lamp;

FIGURE 3 is a graphical representation of the spectral output of an alternative preferred UV lamp; and

~~FIGURE 4 shows the chemical structure of a reactant.~~

Referring initially to Figure 1 of the accompanying drawings, an apparatus for curing a coating applied to a substrate is illustrated.

An apparatus is illustrated which comprises a pair of rollers 1,2 adapted to guide a substrate 3, such as a sheet of aluminium foil or a sheet of paper, through the illustrated apparatus. The substrate 3 is coated, before entering the apparatus, completely or partially, with a curable composition which will be described in greater detail hereinafter. The curable composition may be applied as an un-broken coating or may be applied in the form of printing.

The rollers 1,2 guide the substrate 3 through a channel 4 defined between a cooled backing plate 5, and a

-6-

super-imposed cover 6. The cover 6 supports two lamps 7,8.

The lamp 7 can be a "D" lamp, as supplied by Fusion Inc. The lamp 7 is located above a quartz window 9 that is sealed against the cover 6 so that light from the lamp 7 may shine through the quartz window 9 on to the substrate 3 as it passes through a curing zone formed by the channel 4.

The lamp 8 can be a "H" lamp as supplied by Fusion Inc. and is also associated with a quartz window 10 which is formed integrally with the cover 6 so that light from the lamp 8 can pass through the quartz window 10 into the curing zone in channel 4, and thus on to the substrate 3.

The lamps 7,8 each have a focussing reflector. The position of the lamps may be interchanged. In an alternative embodiment, only one lamp may be used, or two lamps of the same type may be used.

The lamps 7 and 8 emit UV light with wavelengths in the band 200 to 550 nm. The light is directed on to the substrate in a region in the central part of the channel 4.

The channel 4 is flushed with nitrogen in order to ensure that there is an inert atmosphere having a minimum quantity of oxygen within the channel. Thus, nitrogen is introduced through an inlet conduit 11 and passes to a dispersing nozzle 12 known as the "inlet knife" which is located on the under-side of the cover 6 above the roller 1, and which is adapted to prevent oxygen entering the channel 4. The nitrogen flows along the channel 4, past the region where the UV light is directed on to the substrate, in the same direction as the direction of movement of the substrate 3. Nitrogen may also be injected into the channel 4 via nozzles located around the periphery

-7-

of each quartz window. At the end of the channel 4 adjacent the roller 2 nitrogen flowing through a conduit 13 is passed through a nozzle 14 on the underside of the cover into the channel 4. Thus, there is a continuous flow of nitrogen within the channel 4 such that the concentration of oxygen within the channel 4 is less than 1,000 ppm and preferably less than 100 ppm. Instead of using nitrogen, other inert gases could be used. However, nitrogen is preferred because it is the least expensive inert gas.

The lamps 7 and 8 are supplied in a modular form, each module is 25.4 mm wide and extends transversely across the direction of movement of the web 3 through the channel 4. The lamps are high intensity lamps using medium pressure mercury vapour bulbs operating at a power level in excess of 140 watts per linear centimetre, preferably in excess of 180 watts per linear centimetre, and most preferably in the region of 240 watts per linear centimetre.

The output spectrum of the "D" lamp 7 is illustrated in Figure 2. It can be observed that the spectrum has a substantial spectral content within the range of 350-450 nm, with specific peaks at 355, 370, 383 and 408 nm. There is spectral content in the region of 200-350 nm, although the spectral content in the 200-250 nm range is minimal.

The spectrum of the "H" lamp 8 is illustrated in Figure 3. It can be seen that the spectrum has substantial spectral content in the region of 200-300 nm, with the spectral content at the longer wavelengths being restricted to isolated peaks at 312 nm, 370 nm, 408 nm and 438 nm. The spectral content in the region of 200-300 nm comprises a very broad peak centred on 225 nm and spanning the area from 210-240 nm and a further relatively broad peak, which is less clearly defined, but which is substantially centred

-8-

on 262 nm and effectively spans the range of 240-280 nm. It is believed that this very substantial spectral content at these very short wavelengths plays a significant role in creating free radicals to initiate polymerisation. The very high energy present in the well defined peaks at higher frequencies may also contribute.

The reactive part of the curable coating that is applied to the web 3 does not contain a photo-initiator, but does contain a substantial proportion (between 30% and 100% by weight) of multi-functional radiation curable elements. A multi-functional radiation curable element is a radiation curable element which comprises two or more functional groups. Functional groups are acrylate groups with C=C double bonds. If functionality is expressed as a number, the number indicates the number of C=C double bonds available to react, present in acrylate groups.

The radiation curable elements are preferably of low viscosity and can be considered to be reactive diluents, not only providing reactive capabilities, but also maintaining, in the unreacted state, the desired liquid properties of the coating material.

It is preferred that the average molecular weight of any single multi-functional radiation curable element utilised in the curable coating should be greater than 480.

It has been found that relatively low molecular weight radiation curable elements may give rise to skin irritation. It is, however, believed that by utilising a molecular weight greater than 480, the risk of skin irritation arising is substantially reduced or obviated.

Typical examples of multi-functional radiation curable reactive diluents are propoxylated pentaerythritol tetra-

-9-

acrylate or ethoxylated pentaerythritol tetra-acrylate. An alternative material comprises "OTA 480", a triacrylated low viscosity material available from UCB Chemicals of Anderlecht Str. 33, B-1620 Drogenbos, Belgium. The structure of OTA 480 is shown in Figure 4.

The curable coating may optionally comprise, in addition to the reactive part, a non-reactive part or filler that may comprise clay or silica. In some cases, where the coating is to have magnetic properties, the filler may comprise metal particles that may be magnetised.

It is believed that the very high intensity UV radiation applied by the lamps to the curable material generates sufficient free radicals to initiate the curing of the coating.

As the reaction takes place within the substantially inert atmosphere, it is thought that even though a relatively low number of radicals may be produced, because of the high functionality of the coating material, and because the radicals are not subject to oxygen quenching, the radicals that are available to initiate the reaction are sufficient to enable the reaction to proceed very rapidly. Although multi-functional materials, where functionality is greater than 3, are highly reactive, they are believed to undergo a relatively low level of conversion to form a fully cured coating, when compared to mono or di-functional materials.

It is believed that the physical properties of a coating formed solely from multi-functional reactive diluents of low viscosity, whilst sufficient for many purposes, may not be considered sufficient for use as a high performance coating. In order to produce a coating

-10-

formulation that exhibits solvent resistance and stain resistance, it has been found appropriate to combine, with the reactive diluents, a pre-polymer which preferably contains some unsaturation. A pre-polymer is a reactive material of relatively high viscosity. A pre-polymer, when used alone, does not exhibit liquid properties that are appropriate for a coating material that could be used for example in a direct gravure coating process. Examples of suitable pre-polymers are polyester acrylates, polyurethane acrylates and epoxy acrylates. The functionality of these materials is normally 2 or 3 but can be up to 6. It is thought that the higher the functionality of the pre-polymers, the faster the curing performance of the coating.

#### EXAMPLE 1

A series of multi-functional materials were coated on to aluminium foil at a coat weight of approximately  $10\text{g/m}^2$ .

The coated foil was passed through a curing apparatus, similar to that shown in Figure 1, but with only one lamp at a speed of 20 m/min. Initially, the lamp was an "H" lamp, and subsequently the experiment was repeated using a "D" lamp. The lamps were each operated at a power level of 240 watts/centimetre. The cured coating was subsequently tested using an acetone rub test utilising a SATRA rub tester. Such a tester provides an indication of the degree of curing. Solvent resistance is indicated as the number of double rubs effected before the coating applied to the aluminium foil is removed. The greater the number of rubs, the more solvent resistant is the coating.

-11-

The results are set out in Table 1.

TABLE 1

Coating Composition	Solvent Resistance - Acetone rubs	
	H Bulb	D Bulb
<u>Di and Triacrylates</u>		
Tripropan-glycol-diacrylate (UCB)	<2	<2
1,6-hexanediol diacrylate (UCB)	25	<2
OTA 480 triacrylate (UCB)	40	<2
<u>Tetra-acrylates</u>		
Ebecryl 40 (UCB)	95	<2
<u>Ethoxylated-pentaerythritol tetra-acrylate (Croda)</u>	45	<2
<u>Tetra-acrylate with pre-polymer</u>		
Actilane 320 PP 50 Epoxy acrylate with 50% Propoxylated-pentaerythritol tetra-acrylate (Ackros)	150	<2

Ebecryl 40 is a tetra-functional acrylated reactive diluent available from UCB Chemicals. The ethoxylated pentaerythritol tetra-acrylate was obtained from Croda Resins Ltd. of Crabtree Manorway, South Belvedere, Kent DA17 6BA. Actilane 320 PP 50 is obtained from Ackros Chemicals, Eccles Site, Bentcliffe Way, P.O. Box 1, Eccles, Manchester M30 0BH.

-12-

Example 1 shows that when the coating composition comprises only a di-acrylate, the solvent resistance has a very low value. When tri-acrylates or tetra-acrylates are utilised, a much greater degree of solvent resistance is achieved. When a tetra-acrylate mixed with a pre-polymer is utilised, there is substantial improvement in the performance of the cured coating. This is believed to be due to the introduction of higher molecular weight epoxy groups in the cross-linked structure of the coating.

#### EXAMPLE 2

A coating of HH52-0103 02 EBC foil coating material obtained from Glasurit GmbH, Postfach 6123, D-48136 Muenster, Germany, was applied at a coat weight of approximately  $10\text{g/m}^2$  to an aluminium foil. This material comprises a relatively small proportion epoxy acrylate and a relatively large proportion of ethoxylated pentaerythritol tetra-acrylate. The coated foil was passed at various speeds through a curing apparatus similar to that shown in Figure 1 but with only a single "H" lamp 7 operating. In an initial series of experiments, the "H" lamp was operated at a power of 240 watts per centimetre and in another series of experiments the "H" lamp was operated at a power of 160 watts per centimetre. The acetone rub test as described with reference to Example 1, was subsequently used to indicate the degree of curing. The results are shown in Table 2.

**TABLE 2**

Speed	Solvent Resistance Acetone rubs	
	@ 240 W/cm	@ 160 W/cm
30 M/min	100	11
40 M/min	35	9
50 M/min	9	1
60 M/min	4	1

Example 2 illustrates that the degree of curing of the coating is improved when the coating is provided with a substantial energy input in the UV spectrum. The best results are achieved with a relatively low speed of movement of the substrate through the curing apparatus and with the application of very intense high energy light. It can be seen that it is important, for a satisfactory cure to be achieved, for there to be a sufficient power input for a sufficient period of time. The degree of cure achieved does not increase linearly with lower speed of passage through the curing zone (i.e. does not increase linearly with the exposure time to intense UV light). As can be seen, especially at 240 watts per centimetre, the degree of cure achieved increases very substantially, almost exponentially, with increased residence time in the curing zone.

It is believed that an even higher degree of cure can be achieved utilising two lamps within the curing zone. This is shown in Example 3 where the same coating is applied and cured using two lamps at a much higher speed.

EXAMPLE 3

A coating of HH52-0103 02 EBC foil coating material was coated on to a printed paper (Paper 13-2606), using a gravure coating unit at a coat weight of approximately 8 g/m<sup>2</sup> and a run speed of 80 metres per minute. The coated paper was passed through a UV curing apparatus of a type illustrated in Figure 1 flushed with nitrogen gas to as to achieve a residual oxygen level within the curing region of less than 100 ppm. The curing zone was illuminated, through quartz windows, by two high intensity UV lamps, one "H" bulb and one "D" bulb utilising focus-reflectors to direct UV light on to the coating to cure it to form a dry film, the two lamps operating at a power of 240 watts per linear centimetre.

The solvent resistance was greater than 50 acetone rubs (measured as in Example 2).

To test the performance of the coated paper as a furniture surfacing material, a sample of the coated paper was laminated, using aminoplast adhesive, on to a piece of chipboard. Then stain and scratch tests were carried out using the techniques described in ISO 4211. For comparison purposes, the test run was repeated, adding to the coating material the photo-initiator recommended by, and obtainable from, Glasurit GmbH, which is a methyl-phenyl-glyoxylate identified as SR07 641H. This coated paper was also laminated to a piece of chipboard using aminoplast adhesive. For further comparison, a sample of commercially available furniture foil which had been lacquered using a water-based amino polyester lacquer and then thermally

-15-

cured, was also laminated, using aminoplast adhesive, to chipboard to represent a current commercially acceptable industry performance standard.

The results are given in Table 3, where stain resistance is indicated on a 1-5 scale (5 being the best result and 1 being the worst result).

TABLE 3

Test Liquid	ISO 4211 Stain Resistance		
	Thermal Cured	UV No. Photoinitiator	UV + 0.5 % SR07-641H
	Lacquer		
Acetic Acid	3.0	3.0	2.0
Acetone	3.0	3.0	3.0
Ammonia solution	3.0	3.0	3.0
Blackcurrant juice	3.0	3.0	3.0
Citric acid	3.0	3.0	3.0
Cleansing agent	3.5	3.0	3.0
Coffee	3.0	3.5	3.5
Disinfectant (DETTOL)	3.5	4.0	4.0
Disinfectant (SAVLON)	3.0	4.0	4.0
Endorsing ink	4.0	3.0	3.0
Ethanol	3.5	3.5	3.5
Ethyl/Butyl acetate	3.5	3.5	3.0
Iodine	4.0	3.0	3.0
Milk (condensed)	3.0	3.0	3.0
Olive Oil	3.5	3.5	3.5
Paraffin oil BP	3.5	3.5	3.0
SBP Spirit	4.0	3.0	3.0
Sodium carbonate	4.0	4.0	4.0
Sodium chloride	4.0	4.0	3.0
Tea	4.0	4.0	3.5
Water	3.5	3.5	3.0
TOTALS:	72.5	71.0	67.0

-17-

Example 3 indicates that a coating composition in accordance with the present invention, with no photo-initiator, provides a superior performance to a coating composition which does include a photo-initiator. The performance achieved by the material of the present invention is very similar to the performance achieved by the industry-acceptable-standard utilised for comparison purposes. Furthermore, by utilising a preferred combination of UV lamp power, spectrum and reactive materials, the process can operate at higher speeds.

Consequently, it is believed that the present invention provides a method of producing an industry-acceptable material without the use of photo-initiators, but whilst still providing the other advantages of UV curing.

The coating techniques described above have been found to be particularly suitable for applying coatings to flexible papers or films, such as papers or films produced on high speed coating and printing machines. The coatings have been found to be especially valuable for use on surfaces that are found in the home environment. Thus, the coatings may be applied to surfacing materials intended for application to furniture, walls, floors and ceilings. However, it is to be understood that the method described above may be utilised for less demanding coating applications, such as varnishes for books, magazines or record sleeves. The method described above may also be utilised for fabricating coated materials for use in packaging where low odour coatings, or coatings which do not impart a "taste" to products, are of particular value.

-18-

CLAIMS:

1. A method of coating a substrate, the method comprising the steps of applying a coating composition to at least selected areas of the substrate, exposing the coated substrate to ultra-violet light from at least one lamp having a power output of at least 140 watts per linear centimetre in a curing zone, to initiate curing of the coating, the coating composition comprising a mixture including at least a reactive part comprising between 30% and 100% multi-functional material and being photo-initiator-free, including the step of maintaining a substantially inert atmosphere in the curing zone where the substrate is exposed to said ultra-violet light.
2. A method according to Claim 1 wherein the inert atmosphere is obtained by purging the said curing zone with inert gas.
3. A method according to Claim 2 wherein the inert gas comprises nitrogen.
4. A method according to any one of the preceding Claims wherein the oxygen concentration within the said curing zone is less than 1,000 parts per million.
5. A method according to Claim 4 wherein the oxygen concentration is less than 100 parts per million.
6. A method according to any one of the preceding Claims wherein the multi-functional material comprises one or more reactive diluents.
7. A method according to any one of the preceding Claims wherein the multi-functional material comprises one or more

-19-

materials, the or each material having a molecular weight in excess of 480.

8. A method according to any one of the preceding Claims wherein the multi-functional material comprises one or more materials which have three or more functional acrylate groups.

9. A method according to Claim 6, 7 or 8 wherein the coating material additionally contains a pre-polymer.

10. A method according to Claim 9 wherein the pre-polymer comprises polyester acrylate, polyurethane acrylate, epoxyacrylate, or a full acrylate material.

11. A method according to Claim 9 or 10 wherein the pre-polymer is multi-functional.

---

12. A method according to any one of the preceding Claims wherein the coating composition comprises, in addition to the reactive part, a filler.

13. A method according to Claim 12 wherein the filler is clay.

14. A method according to Claim 12 wherein the filler is silica.

15. A method according to Claim 12 wherein the filler is magnetisable particles.

16. A method according to any one of the preceding Claims wherein the power output of the lamp is at least 180 watts/cm.

-20-

17. A method according to Claim 16 wherein the power output of the lamp is substantially 240 watts/cm.

18. A method according to any one of the preceding Claims wherein UV light from the lamp has a substantial spectral content in the range of 200-300 nm.

19. A method according to Claim 18 wherein UV light from the lamp has a spectral content at peaks of approximately 370 nm, 408 nm and 438 nm.

20. A method according to any one of the preceding Claims wherein two lamps are provided in the curing zone, the lamps having different spectral properties.

21. A method according to any one of Claims 1 to 19 wherein two lamps are provided in the curing zone, the lamps having substantially identical spectral properties.

22. A substrate when coated by a method according to any one of the preceding Claims.

23. A method of coating a substrate substantially as herein described by way of example.

1 / 3

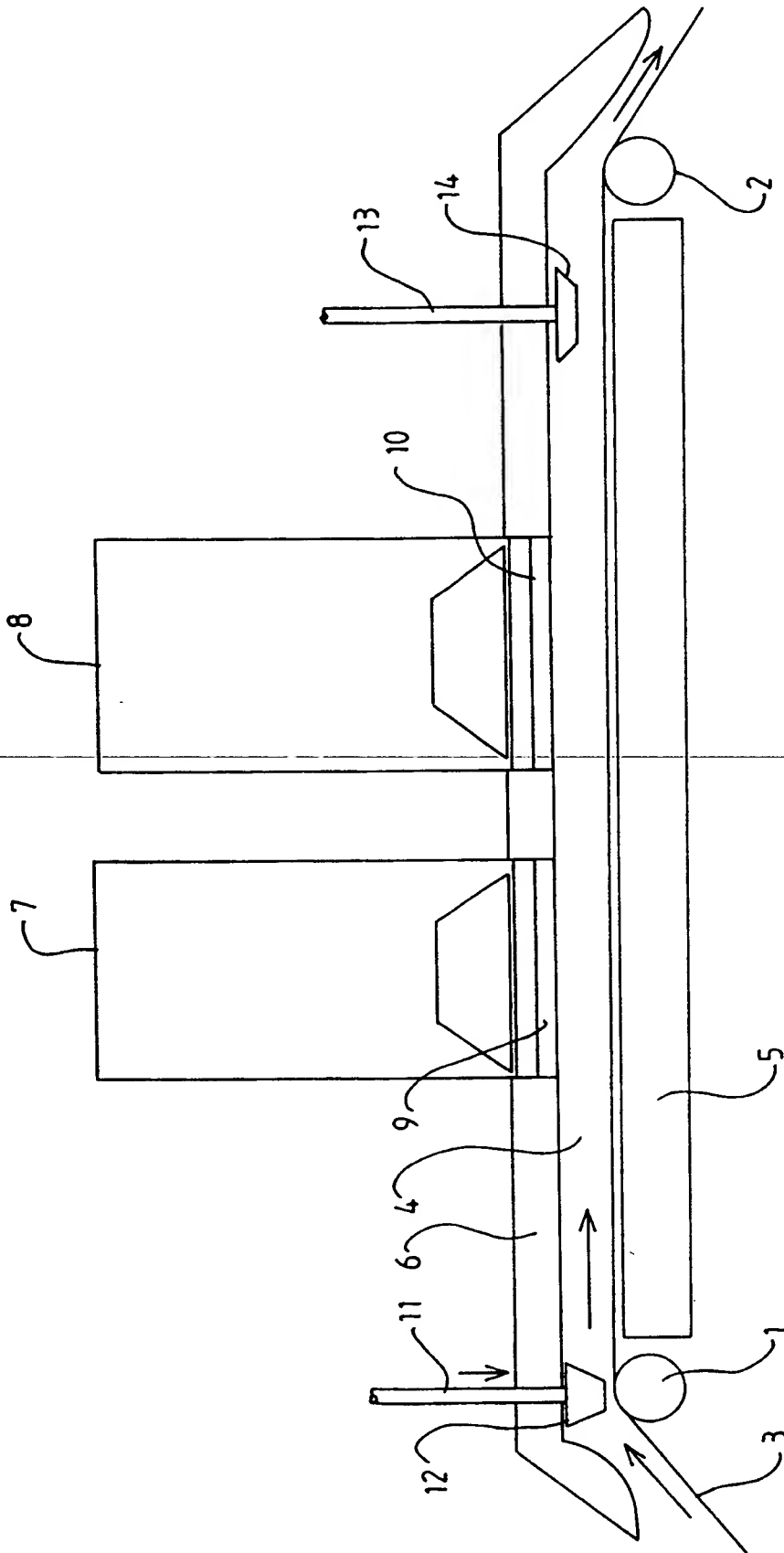
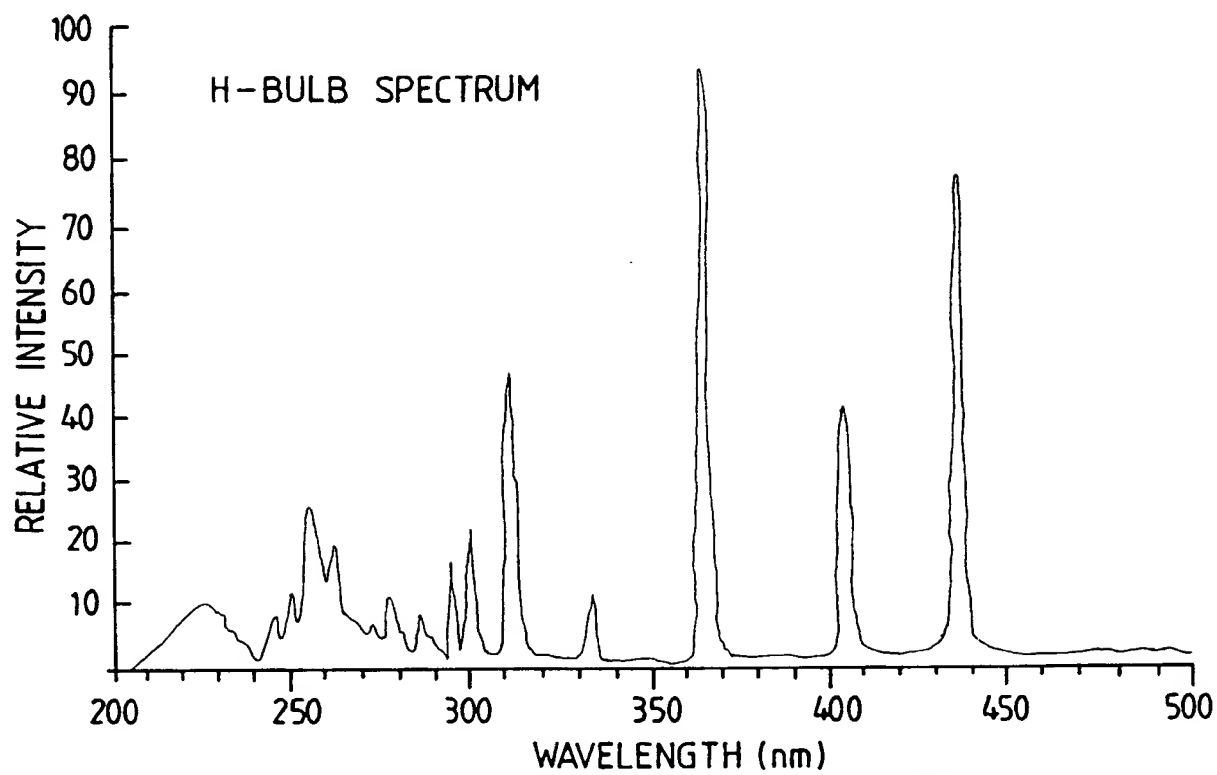
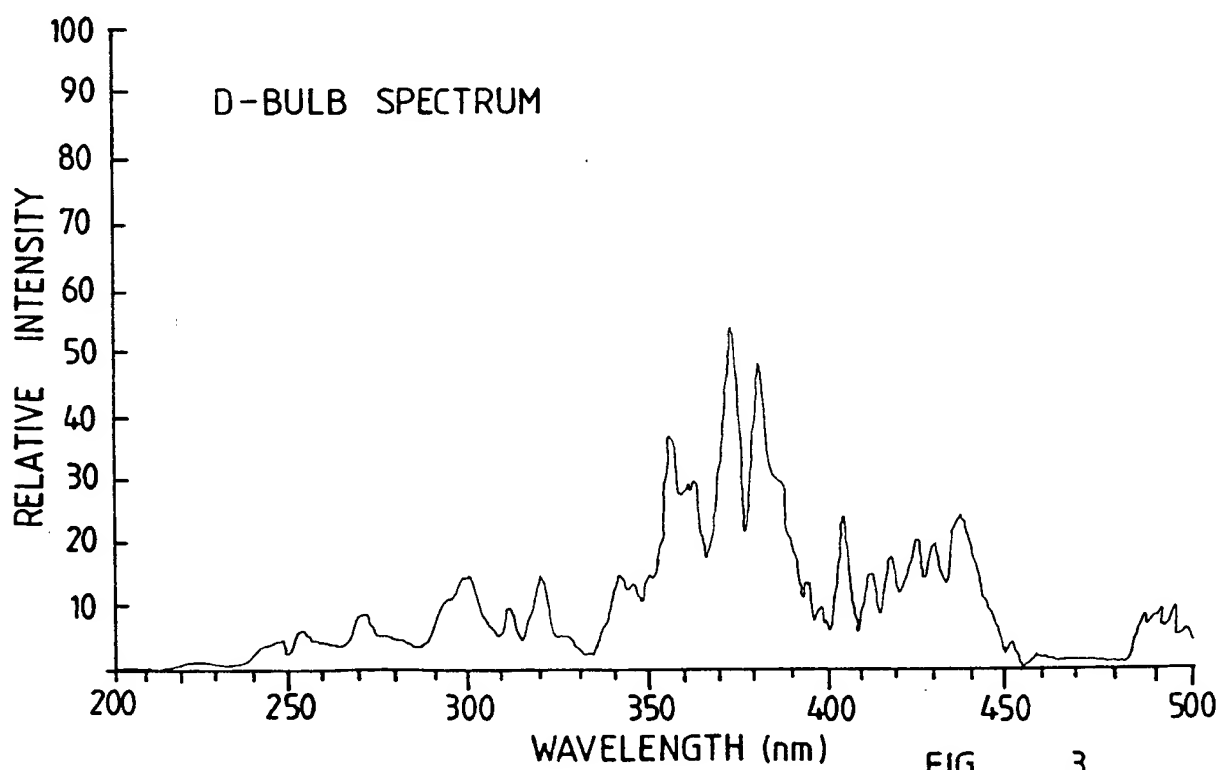
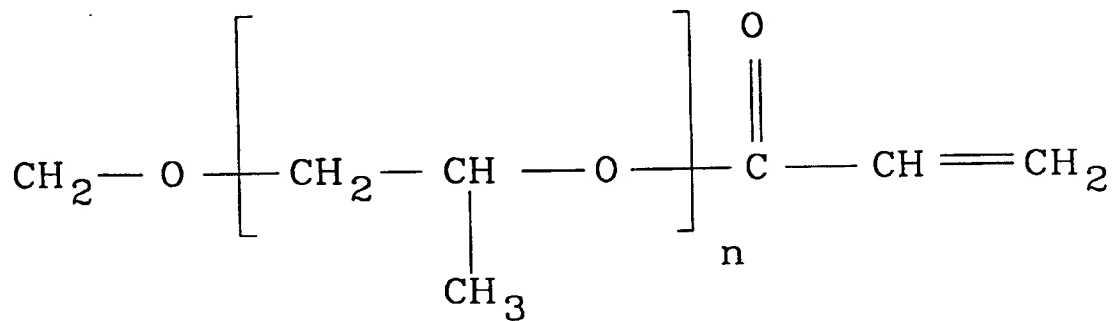
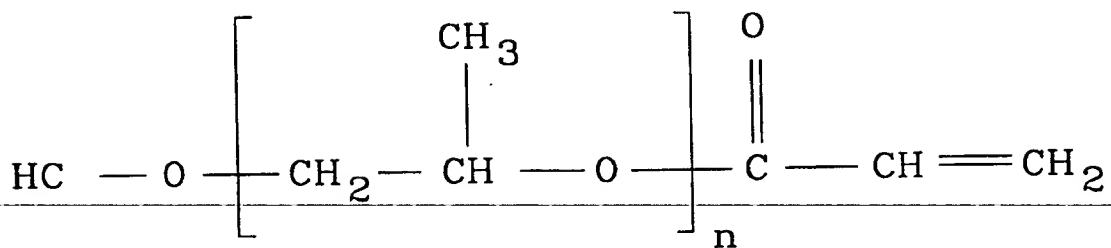
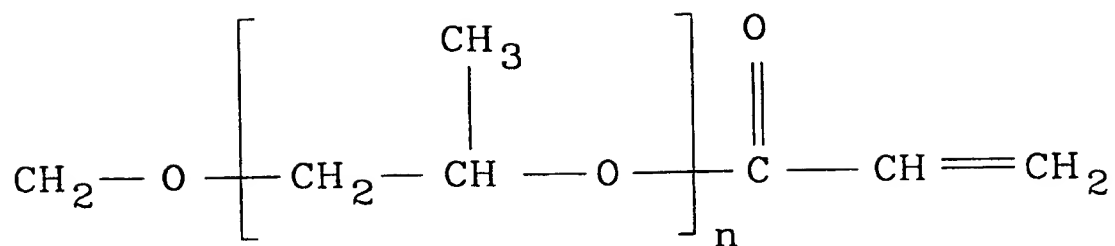


FIG 1

2 / 3

FIG 2FIG 3

3 / 3

FIG 4

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 98/01309

## A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C09D 4/00 // C08F 2/48

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C08F, C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5047261 A (KHALIL MOUSSA ET AL), 10 Sept 1991 (10.09.91), column 2, line 3 - line 45, claims 1, 8 --	1-23
A	US 5446073 A (SONNY JONSSON ET AL), 29 August 1995 (29.08.95), abstract --	1-23
A	US 5188900 A (ANTHONY REVIS ET AL), 23 February 1993 (23.02.93), column 3, line 40 - column 4, line 9, claim 1 --	1-23
A	US 4557975 A (JAMES E. MOORE), 10 December 1985 (10.12.85), claim 1 --	1-23

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

\* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&amp;" document member of the same patent family

Date of the actual completion of the international search

5 November 1998

Date of mailing of the international search report

06 - 11 - 1998

Name and mailing address of the ISA/

Swedish Patent Office

Box 5055, S-102 42 STOCKHOLM

Facsimile No. +46 8 666 02 86

Authorized officer

Barbro Nilsson

Telephone No. +46 8 782 25 00



# INTERNATIONAL SEARCH REPORT

Information on patent family members

05/10/98

International application No.

PCT/SE 98/01309

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5047261 A	10/09/91	DE 69004625 D,T EP 0406057 A,B FR 2649111 A,B IE 63812 B JP 3047567 A	26/05/94 02/01/91 04/01/91 14/06/95 28/02/91
US 5446073 A	29/08/95	AT 170534 T EP 0618237 A,B JP 6298817 A	15/09/98 05/10/94 25/10/94
US 5188900 A	23/02/93	CA 2027321 A DE 69024316 D,T EP 0424006 A,B ES 2084007 T JP 3140371 A US 5126394 A	19/04/91 18/07/96 24/04/91 01/05/96 14/06/91 30/06/92
US 4557975 A	10/12/85	NONE	
EP 0549116 A2	30/06/93	SE 0549116 T3 AT 163021 T DE 69224365 D,T ES 2113415 T US 5234970 A	15/02/98 10/09/98 01/05/98 10/08/93